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TITLE **SHOCK RECOVERY EXPERIMENTS: AN ASSESSMENT**

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SHOCK RECOVERY EXPERIMENTS: AN ASSESSMENT

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Systematic shock recovery experiments, in which microstructural and mechanical property effects are characterized quantitatively, constitute an important means of increasing our understanding of shock processes. Through studies of the effects of variations in metallurgical and shock loading parameters on structure/property relationships, the micromechanisms of shock deformation, and how they differ from conventional strain rate processes, are beginning to emerge. This paper will highlight the state-of-the-art in shock recovery of metallic and ceramic materials. Techniques will be described which are utilized to "soft" recover shock-loaded metallic samples possessing low residual strain; crucial to accurate "post-mortem" metallurgical investigations of the influence of shock loading on material behavior. Illustrations of the influence of shock assembly design on the structure/property relationships in shock-recovered copper samples including such issues as residual strain and contact stresses, and their consequences are discussed. Shock recovery techniques used on brittle materials will be reviewed and discussed in light of recent experimental results. Finally, shock recovery structure/property results and VISAR data on the α–ω shock-induced phase transition in titanium will be used to illustrate the beneficial link between shock recovery and "real-time" shock data.

1. INTRODUCTION

The study of the structure/property relationships in materials while under shock loading pressures is very difficult due to the dynamic nature of the shock process and the very short time available for study. Due to these imposed constraints, most "real-time" shock process measurements are limited to studying the interactions of the transmitted waves upon arrival at the free surface. To augment these in-situ wave-profile measurements, shock recovery techniques were developed in the late 1950's to experimentally assess the residual effects of shock wave compression on materials.

The object of "soft" recovery experimentation is to examine the terminal structure/property relationships of a material that has been subjected to a known uniaxial shock history, i.e. controlled peak pressure and pulse duration, and has been returned to ambient pressure <u>without</u> experiencing radial release tensile wave loading or collateral recovery strains. Tensile wave interactions may be mostly mitigated by surrounding the sample with tightly fitting material of the same or nearly the same shock impedance, both laterally and below the sample. This technique, termed "momentum trapping" has continued to evolve to prevent radial release rarefactions and spallation from entering the sample in a variety of sample configurations and for various shock loading methods. When ideally trapped, the residual strain (ε_{res}) in the sample (defined here as the final sample thickness divided by the initial sample thickness) should be on the order of only a few percent. Since the inception of shock recovery studies, the use of momentum trapping techniques has been successfully applied to a large number of metallic systems and a more limited number of brittle solids. Several review papers have chronicled the development of shock recovery techniques employing gas guns, exploding wires, or explosive loading to shock load materials.[1-4]

This paper will briefly review the current state-of-the-art

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This paper will briefly review the current state-of-the-art and future frontiers of shock recovery experimentation of metals and ceramics. Recently quantified factors influencing "soft" shock recovery, including the importance of accurately limiting radial release interactions resulting in residual strains and their effects on subsequent structure/property relationships, will be discussed. Shock

recovery techniques used for brittle solids such as ceramics, intermetallics, and minerals will be compared/contrasted with recovery techniques used for metallic materials. Finally, the advantages of performing "soft" shock recovery techniques in parallel with "real-time" shock wave studies will be illustrated.

2. BACKGROUND

The extremes in the loading path during a shock induce a high density of defects in most materials, i.e., dislocations, point defects, and/or deformation twins.[1,2,5-7] In addition, during the shock process some materials may undergo a pressure-induced-phase transition which will affect the "real-time" material response and if the phase remains present to ambient conditions, although metastable, the post-mortem substructure and mechanical response will reflect the high pressure excursion. Interpretation of the results of shock recovery experiments must therefore address all of the details of the shock-induced deformation substructure in light of the operative metallurgical strengthening mechanisms in the material under investigation.

Several in-depth reviews have summarized the micro-structural and mechanical response literature on shock recovered metals and alloys.[1,2,5-7] Overall, the deformation substructures resulting at modest shock pressures (0 - 40 GPa) in metals from shock-induced defects are observed to be very uniformly distributed on a grain to grain scale. The specific type of substructure developed in the shock in a given metal, i.e., dislocation cells, twins, faults, etc., has been shown to critically depend on a number of factors including the crystal structure of the metal or alloy, the relevant strengthening and deformation mechanisms in the material studied (such as alloying, grain size, second phases, interstitial content, etc.), temperature, stacking fault energy, and the shock parameters. The overall substructure, while

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microstructural changes in metallic systems in turn correlate with variations in the "post-mortem" mechanical properties, with increasing peak shock pressure in particular, leading to increases in both the hardness and reload yield strength.[1,2,5-7] Shock loading in most metals and alloys produces greater hardening than quasi-static deformation to the same total strain, particularly if the metal undergoes a polymorphic phase transition, such as pure iron.[1,2,5-7] This phenomena has been attributed to the very high strain rates associated with shock loading and the subsonic restriction on dislocation velocity requiring the generation of a larger dislocation density during the shock process than for quasi-static processes[1,2] and the rate dependence of dislocation storage.[8] While this concept can qualitatively be applied to explain the ~ six fold yield increase in copper subjected to a 10 GPa shock[9], significant shock hardening is not observed in all metals. In some alloys such as 6061-T6 Al or Ti-6Al-4V shock loading is observed to cause minimum shock strengthening with the post-shock yield strength nearly equivalent to the quasi-statically deformed material yield.[10,11] These shock-loading results suggest that in the specific cases of 6061-T6 Al (where quasi-statically there is minimal strain hardening, i.e. dislocation storage during deformation) and Ti-6Al-4V there is not a significant strain-rate dependence of the strain-hardening as contrasted to shock-loaded copper or many other metals.

3. RADIAL RELEASE EFFECTS

The residual properties and microstructure observed in shock recovered samples have been tacitly assumed to result solely from the compression and unloading due to the imposed uniaxial-strain shock. Recent shock recovery studies have however shown that the degree of residual strain ε_{res} in the sample on examination significantly influences the measured structure/property relationships and can even over-shadow the shock wave parametric

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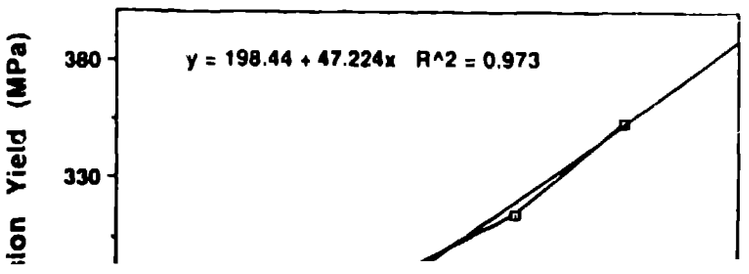
Two-dimensional finite difference calculations on 6061-T6 aluminum by Stevens and Jones have shown that low ε_{res} values require the careful design of shock recovery assemblies utilizing momentum trapping rings to mitigate radial release[14]. Their modeling results showed that under very unfavorable circumstances the additional plastic work, input to the sample from radial release waves, can be up to 1000% of the plastic work produced by the uniaxial shock process itself[14]. Their study also identified the importance of increasing the diameter-to-thickness ratio of the sample to greater than 7 to 1 to additionally reduce radial release effects.

To further examine the affect of radial release interactions on the structure/property relationships in shock loaded materials, experiments were conducted on copper shock-loaded using several shock-recovery designs which yielded differences in ε_{res} while all having been subjected to a 10 GPa, 1 μsec pulse shock[9]. Copper samples were shock-loaded using an 80-mm gas gun , "soft" recovered, samples were sectioned from the recovered sample to measure the reload yield behavior, and samples were examined in the transmission electron microscope(TEM) to study the substructure evolution. Details of the experimental set-up and shock recovery design parameters are presented in-depth elsewhere[9].

The substructure and yield strength of the bulk shock-loaded copper samples were found to depend on the amount of ε_{res} in the shock-recovered sample at a constant peak pressure and pulse duration. In Figure 1 the reload yield strength of the 10 GPa shock-loaded copper is observed to increase with increasing residual sample strain.



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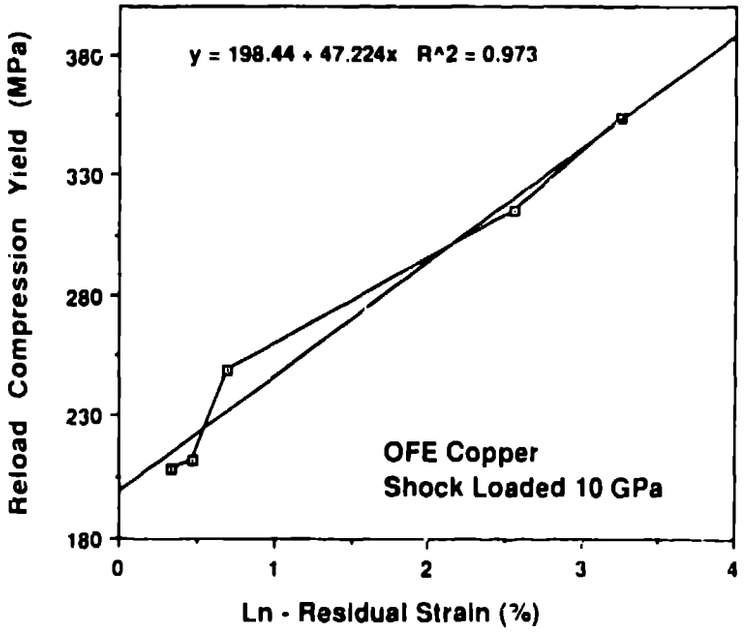

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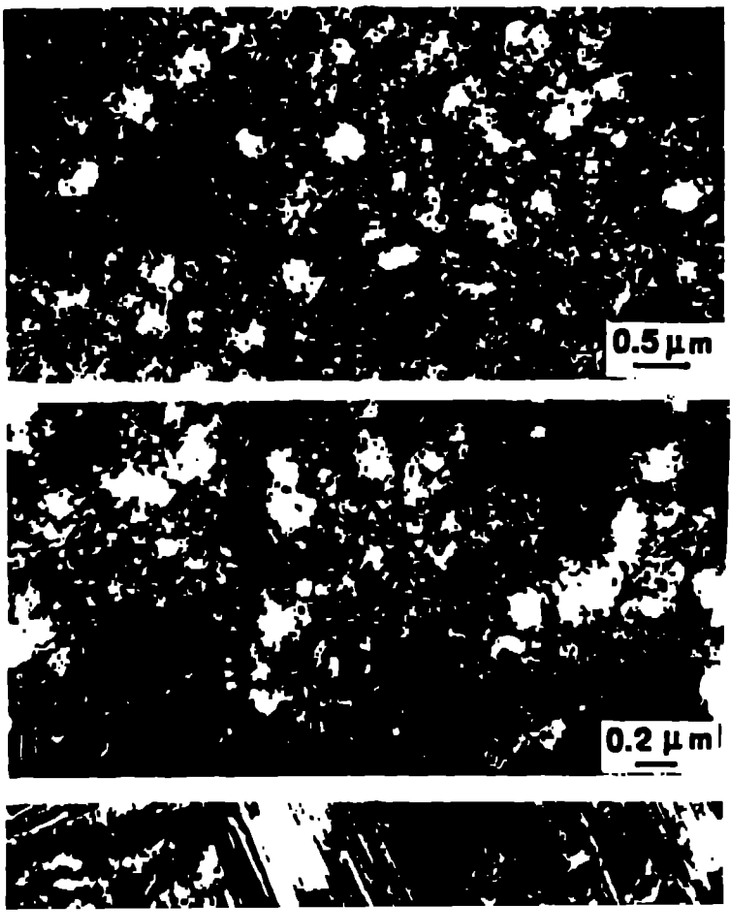
FIGURE 1

Reload Yield Strength vs. Residual Strain for Shocked Copper

TEM examination showed that the substructure of the shock-loaded copper consisted of almost entirely dislocation cells at low ε_{res}. With increasing ε_{res} the substructure became more heterogeneous in nature, with grains exhibiting planar slip bands, microbands, and deformation twins intermixed with dislocation cells. Figure 2 shows typical TEM bright-field micrographs of the substructure of shock-loaded copper as a function of ε_{res}. At the 26% ε_{res} level, nearly all grains contained deformation twins, with the twins most often occurring in packets composed of many fine twins.



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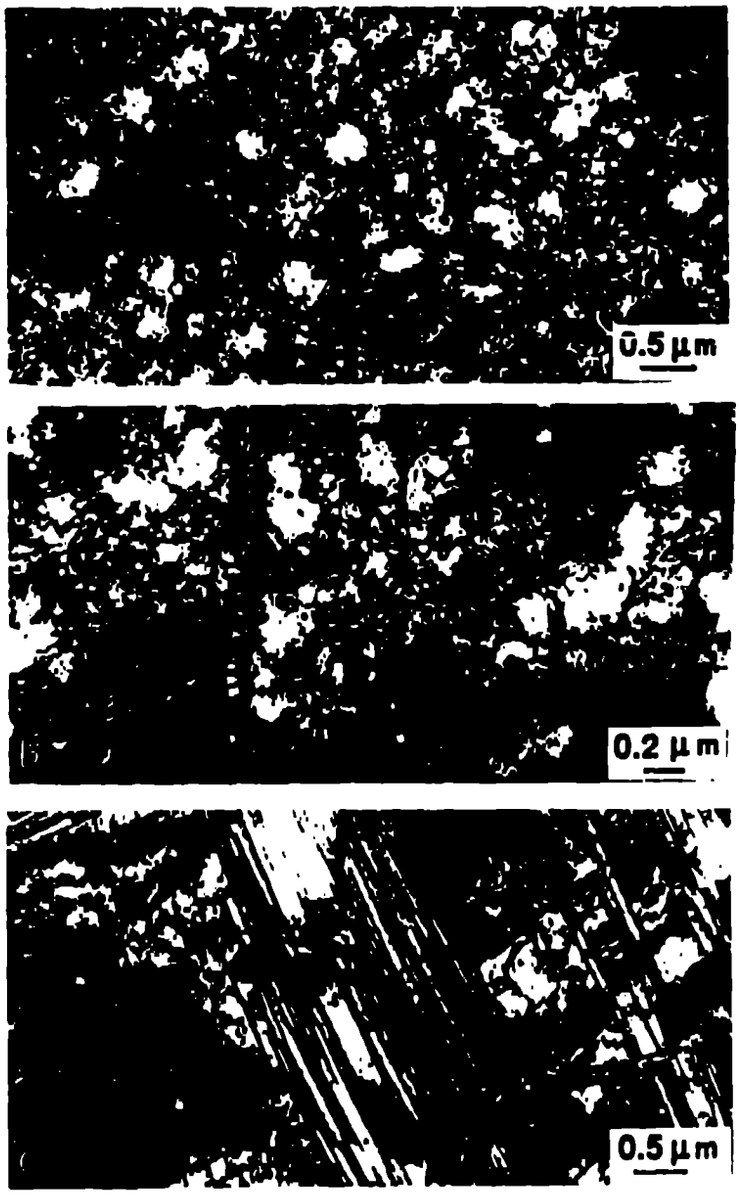

FIGURE 2

TEM micrographs of shock-loaded copper as a function of ε_{res}: a) <2% ε_{res}, b) 7% ε_{res}, c) 26% ε_{res}

The increased reload mechanical response and the development of increasing amounts of planar slip, microbands, and deformation twins with increasing ε_{res} is thought to reflect increased applied shear stresses from "late-time" loading, i.e., post-uniaxial-shock compression and release. This loading is due to insufficient momentum trapping leading to radial release wave unloading of the sample. The effectiveness of this unconstrained radial plastic flow on strain hardening is readily apparent from the fact that the 26 % ε_{res}, 10 GPa sample possesses a reload yield strength (350 MPa) which exceeds the yield of a low ε_{res} 20 GPa shocked sample (286 MPa) by nearly 20%[9].

The substructure evolution sequence of planar slip bands appearing in addition to a dislocation cell structure with increasing ε_{res} in this study is identical to that observed in quasi-static studies of the effects of stress path changes on dislocation substructure development[15]. In quasi-static studies on the influence of stress path change on substructure evolution in copper, deforming a sample in tension at 90 degrees orthogonal to the previous compression axis was observed to cause coarse planar slip band formation cutting across dislocation cells formed during the initial compression excursion. The formation of the planar slip bands across the pre-existing cell substructure has been related to the instability of resident substructures to stress path changes altering the active slip planes[15]. Upon altering the stress path 90 degrees after a preliminary deformation on specific slip systems, different slip planes are activated[15].

Initially, the dislocations which move most probably originate in the cell walls and after traversing the cell interior begin to interact with the forest dislocations in the next cell wall. With increasing uniaxial strain, positive and

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slip concentration across portions of the crystal which develop into coarse slip bands[15]. The appearance of planar slip bands in addition to the dislocation cells with moderate increases in ε_{res} in this study, as seen in Figure 2b, is thought to be the result of a similar mechanism. In the present shock-loading case the stress path change is the result of "late-time" radial release wave effects applied 90 degrees to the pre-existing uniaxial-strain shock-formed cell substructure. Further increases in the ε_{res} will result in addition plastic work locally increasing the applied shear stresses to levels capable of activating widespread microbands and deformation twinning, consistent with this study[9].

4. SHOCK RECOVERY OF BRITTLE MATERIALS

While the structure/property behavior of numerous shock recovered metals and alloys has received considerable attention in the literature to date, the response of ceramics and cermets to shock-loading remains poorly understood. The majority of shock-recovery studies on brittle materials have concentrated on examining the response of minerals and a few monolithic ceramics[2,16]. Several microstructural studies of shock-recovered minerals, such as quartz, biotite, anorthite, and periclase have revealed the formation of several types of planar substructural features, labeled kink bands or "deformation lamellae", both suggesting inhomogeneous plastic flow processes above the Hugoniot Elastic Limit (HEL) in these brittle solids. Conversely, TEM examination of shock recovered olivine found no evidence of "shear bands" or zones which display features most likely generated at high temperatures[17].

Several recent shock-recovery studies have utilized the TEM to examine the substructure evolution in several monolithic ceramics and a cermet[18-21]. A substructural study on shock-recovered fragments of Al_2O_3, TiB_2, and SiC monolithes concluded that for most ceramics, plastic deformation, specifically dislocation generation, under

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examination of the alumina revealed that only selective grains displayed evidence of plastic deformation, with the deformation consisting solely of dislocation debris and no twinning or cracking. In this study[19] the authors noted that the grains displaying dislocation activity were predominantly adjacent to residual pores in the material. TEM observations of shock-recovered Al-B_4C cermets have similarly shown that at shock pressures significantly above the probable HEL, based on an estimated U_s-U_p, of the cermet the sample remains largely intact and microcrack-free[20,21]. Above the HEL the B_4C constituent responds plastically in a reasonable non-uniform (on the grain to grain level) manner[20,21]. Evidence of both dislocations and deformation twinning have been observed in the B_4C grains following a ~10 GPa shock in the 65% B_4C cermet. The intact natures of the shock-recovery studies on alumina[19] and the Al-B_4C cermets[20,21] above the HEL are contrary to earlier recovery tests on polycrystalline alumina, encapsulated in copper or aluminum, which displayed significant fragmentation below the HEL[22].

The lack of extensive data in the literature on fragmentation behavior of shock-loaded brittle solids is thought to be due to: 1) the experimental difficulties associated with shock recovery and 2) the poor success of many techniques utilized to "soft" recover brittle solids. Mineral and ceramic recovery techniques have largely been modifications of successful assembly designs applied to metallic systems. The ceramic or mineral of interest has often been placed in a metallic capsule or threaded container which is in turn surrounded by metallic momentum trapping and backed by a metallic spall plate. The containment of the ceramic or mineral is intended to limit fragmentation and collateral damage on deceleration following the uniaxial shock event. The principal difficulty of the metallic capsule technique lies in the fact that the container is <u>not</u> exactly impedance matched with the brittle solid of interest. The relatively high shock velocity of the ceramic sample compared to the metallic

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sample.

In addition to the metallic capsule impedance problem, the presence of different phases, such as the glassy grain-boundary phase present in some aluminas, and porosity within many ceramics themselves may actually cause significant internal radial release mismatch problems leading to cracking. Accordingly, when such shock recovery techniques or materials yield highly fragmented samples, it becomes impossible to unambiguously ascertain whether the plastic deformation observed in the recovered samples are solely the result of the uniaxial shock process or products of the tensile radial release fragmentation process which occurs subsequent to the planned shock event. These results further show that since radial release interactions in the shock recovery tests may be the primary cause of local fracture in the ceramic or mineral, assignment and/or quantification of the post-recovery micro-cracking behavior of the ceramic is of minimal value.

The recent ceramic recovery experiments which yielded intact sample pieces demonstrate the end effect of mitigating radial release affects[19-21]. Mitigation of the major radial release interactions either through the use of star-shaped flyer configurations[19] in alumina or the use of internal ceramic momemtum trapping rings with the cermet[20,21] drastically reduced fragmentation of the shock-loaded recovery samples. A classic shock recovery technique study on NaCl and KBr illustrated the differences between shock loading a brittle solid in a metallic capsule, which yielded highly cracked recovered samples, and momentum trapping the sample in identical surrounding material which proved very effective[23]. The intact ceramic and cermet recovery experiments[19-21] further dispute the concept that a significant portion of the fragmentation of ceramics may occur during the compression portion of the shock cycle. In fact the recovered alumina (of several different porosity levels) experimental results show that shock-wave deformation

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The differences in the overall fragmentation response of monolithic ceramics and the Al-B_4C cermet in impedance

protected assemblies suggest that the cermet aluminum phase very effectively suppresses micro-cracking and fragmentation in the B_4C skeleton structure. It is thought that the low HEL and very ductile nature of the pure aluminum phase helps laterally confine the B_4C skeleton which prevents axial microcracking and also damps radial release effects transferred to the ceramic phase.

5. SHOCK RECOVERY & "REAL-TIME" STUDIES

Systematic "soft" shock recovery experiments, in which microstructural and mechanical property effects are characterized quantitatively, are increasing being applied to provide an important diagnostic link between "real-time" shock measurements and material behavior. Shock recovery in this context provides a "post-mortem" snap-shot of the structure/property response of a material to the extreme conditions of strain rate, hydrostatic pressure, and temperature imposed by the shock for comparison with wave-profile and shock-reload data.

One illustration of the beneficial teaming of recovery and real-time studies is found in the area of shock-induced phase transformation studies where phase structure can be utilized to corraborate real-time diagnostic data. For example, in the titanium system while the response of titanium alloys to dynamic loading is beginning to be understood, little experimental data exists concerning the structure/property relationships of titanium and titanium alloys subjected to shock loading. These studies are complicated by the fact that pure titanium undergoes a polymorphic transition from the hexagonal α to a more open hexagonal ω phase at high pressure[24]. The ω phase in pure Ti ,formed under either shock or hydrostatic soaking conditions, exhibits a large hysteresis that is responsible for retention of the high-pressure ω phase to atmospheric conditions[24,25].

To investigate the α–ω phase transformation in more detail, parallel "soft" shock-recovery studies and VISAR

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Systematic "soft" shock recovery experiments, in which microstructural and mechanical property effects are characterized quantitatively, are increasing being applied to provide an important diagnostic link between "real-time" shock measurements and material behavior. Shock recovery in this context provides a "post-mortem" snap-shot of the structure/property response of a material to the extreme conditions of strain rate, hydrostatic pressure, and temperature imposed by the shock for comparison with wave-profile and shock-reload data.

One illustration of the beneficial teaming of recovery and real-time studies is found in the area of shock-induced phase transformation studies where phase structure can be utilized to corraborate real-time diagnostic data. For example, in the titanium system while the response of titanium alloys to dynamic loading is beginning to be understood, little experimental data exists concerning the structure/property relationships of titanium and titanium alloys subjected to shock loading. These studies are complicated by the fact that pure titanium undergoes a polymorphic transition from the hexagonal α to a more open hexagonal ω phase at high pressure[24]. The ω phase in pure Ti ,formed under either shock or hydrostatic soaking conditions, exhibits a large hysteresis that is responsible for retention of the high-pressure ω phase to atmospheric conditions[24,25].

To investigate the α–ω phase transformation in more detail, parallel "soft" shock-recovery studies and VISAR experiments have been conducted on high purity titanium and Ti-6Al-4V[26]. Symmetric impact wave-profile shots at a variety of pressures have shown that electrolytic Ti undergoes a phase transition at 10.4 GPa while the Ti-6Al-4V exhibits a linear U_s -U_p response up to 40 GPa[26]. Figure 3 shows a VISAR wave-profile for electrolytic

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titanium shock-loaded to 15.6 GPa.

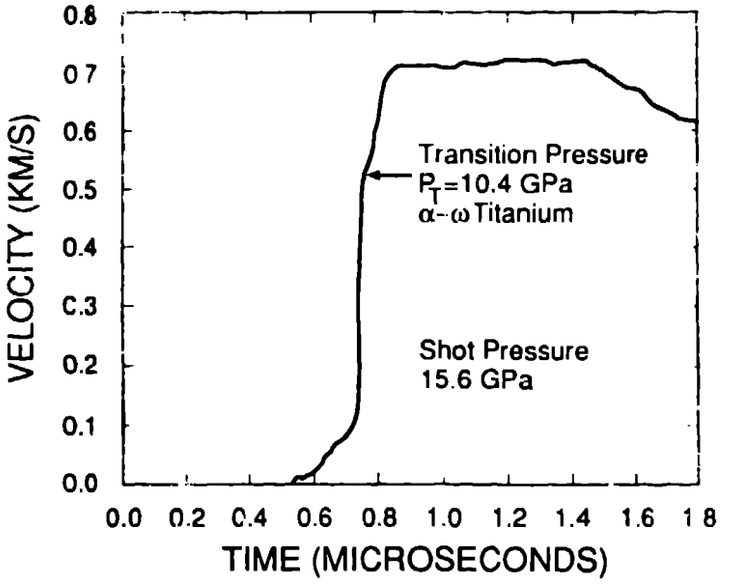
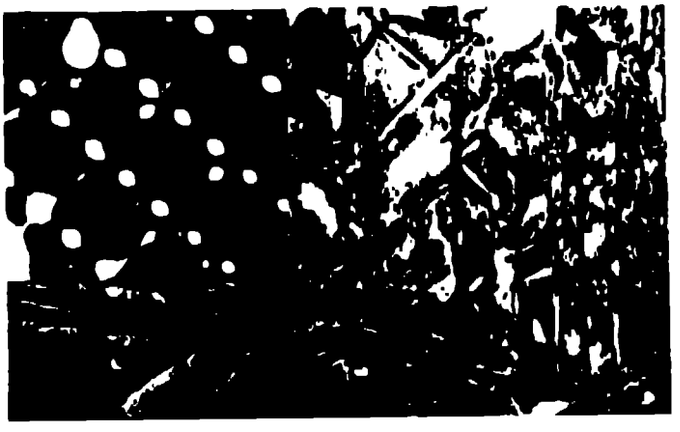


FIGURE 3

Wave-Profile of Shock-Loaded Ti showing the α–ω phase transition.

Bulk X-ray diffractometry and TEM analysis of electrolytic-Ti shock loaded at room temperature, in a wholly Ti shock assembly, to 11 GPa and "soft" recovered (ε_{res} =0.5 %) confirmed the presence of retained ω phase. Bulk x-ray identification of the ω-phase was found to be very sensitive to the sample surface preparation with careful polishing required to avoid mechanical reversion of the ω at the surface. Figure 4 shows a TEM bright-field micrograph and diffraction pattern of the retained ω phase.



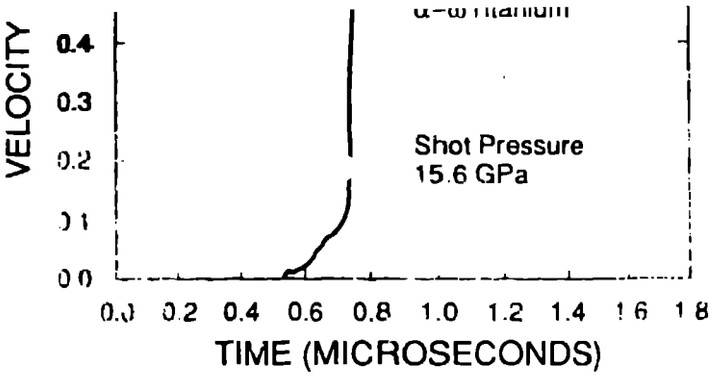


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FIGURE 4

TEM Micrograph of retained ω-phase in"soft" recovered pure Ti.

The current shock-recovery findings are contrary to a previous study where shock loading pure Ti at room temperature to pressures of 12 to 50 GPa yielded no retained ω while finding retained ω if shock loading was conducted at 120K[25]. Shock recovery experiments in the study mentioned[25] were conducted by shock loading the Ti in steel containers which may have influenced the ϵ_{res} and thermal history in the recovered samples. Even in the 120K Ti shots[25], measurement of the phase distribution through the recovered sample thickness revealed the absence of ω at the near impact and rear sample surfaces while showing a uniform amount of retained phase in the sample interior. Due to the mechanical and thermal metastability of the ω-phase, it is believed that the ϵ_{res} in the sample, surface contact stresses[9], and thermal history recovery effects caused in the steel container recovery tests resulted in reversion of the ω during release and deceleration at room temperature in the previous study. The variations in the phase-retention results again graphically illustrate the importance of utilizing "soft" recovery techniques to accurately assess shock-induced structure/property relationships and thereby provide post-mortem physical data for comparison with real-time wave profile data.

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6. FUTURE SHOCK RECOVERY FRONTIERS

Post-mortem characterization of shock-loaded materials will continue to offer valuable data to contribute to the understanding of real-time wave profile and shock wave data. The direction of these studies will however evolve from the traditional emphasis of conducting stand-alone shock recovery materials studies on a given material to the multidisciplinary investigation of a materials total response by integrating shock-recovery techniques with various real-time experiments including wave profile data.

Experimental innovations, such as the PVDF gauge, will assist this combination by allowing the collection and verification of wave-profile data simultaneously with a recovery test. Through a cooperative approach, researchers can strive to establish an in-depth correlation between microstructure and shock-wave response. Such a union is ultimately necessary to developing physically based models capable of predicting and inputing design ideas to allow the control of material response to large-strain, high-strain-rate impulses as a function of stress, stress-path history, and temperature.

The materials chosen for these types of integrated research programs will continue to examine pure metals to facilitate the study of parametric shock-wave studies but also expand their emphasis into the areas of "advanced" engineering materials, such as composites and intermetallics, and brittle solids, both ceramics, cermets, and geological materials. While these new materials are significantly more complicated than single-phase-pure metals, it is imperative that shock-wave research begin to systematically investigate the additive and synergistic nature of multiple strengthening mechanisms on material response to shock-wave deformation. Correlating the shock-induced mechanical response of multi-phase materials, such as composites and cermets, with the wave-profile data will require the development of more sophisticated equation of state models to address multi-phase systems and ordered compounds.

7. SUMMARY

Systematic "soft" shock recovery experiments which quantitatively characterize the structure/property effects of shock deformation continue to provide an important materials view into the shock process. Studies of the influence of shock wave parameters and strengthening mechanisms on the structure/property behavior of shock-recovered materials are however only relevant if the effects

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Systematic "soft" shock recovery experiments which quantitatively characterize the structure/property effects of shock deformation continue to provide an important materials view into the shock process. Studies of the influence of shock wave parameters and strengthening mechanisms on the structure/property behavior of shock-recovered materials are however only relevant if the effects of additional plastic work due to lateral release or poor recovery techniques are minimized as much as possible. This is particularly crucial in brittle materials were significant late-time fragmentation of the material makes it difficult at best to unambiguously ascertain the influence of the shock process on deformation. Future studies

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investigating shock deformation will require interdisciplinary research teams, integrating materials and real-time wave-profile experiments, to understand and model the effects of shock processes on material response and visa versa.

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